

Temperature-modulated DSC (TM-DSC)

Traditional DSC shows phase transitions, specific heat and chemical reactions in segments with linear heating and cooling rates or in isothermal temperature steps with high sensitivity and reproducibility. By varying the heating rates (cooling rates) in consecutive experiments, the sensitivity for individual thermal effects and sometimes also for the separation of overlapping effects can be influenced.

For temperature-modulated DSC, on the other hand, the heating rates are varied by overlapping the underlying linear heating rates with a sinusoidal temperature modulation. The sample to be investigated is almost in

equilibrium due to a linear rate which is mostly low; thermodynamic properties and transitions can therefore be accurately determined. At the same time, the sample is subjected to a non-linear temperature change which is relatively fast in comparison with the linear rate. Not only temperature-dependent but also time-dependent (kinetic) processes in the sample can therefore be detected with sufficient sensitivity.

Figure 1 depicts the simplest evaluation of a typical TM-DSC measurement, which is determination of the total heat-flow curve as an average value of the modulated heat-flow differential curve.

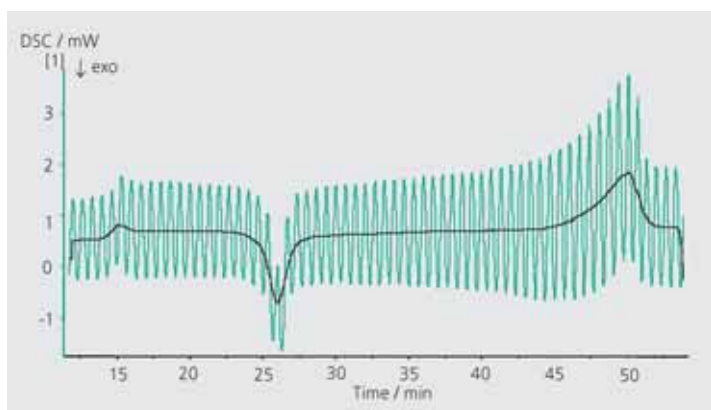


Fig. 1. Modulated signal (green curve) and average values (black curve: total heatflow) of a PET sample

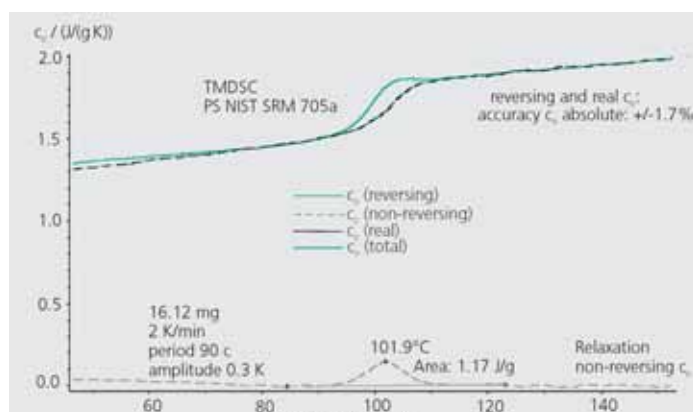


Fig. 2. Determination of c_p and relaxation enthalpy of a freely cooled atactic polystyrene sample (NIST SRM 705 a)

The main application fields of TM-DSC are research and product development. From both a scientific and technological point of view, particularly interesting application areas are the separation between glass transition and overlapping enthalpy relaxation or the separation between glass transition, melting and thermally induced cross-linking in one experiment.

As shown in figure 2 with the example of polystyrene (NIST standard reference material 705 a), both the exact c_p from the reversing signal (or real part of the complex heat capacity) and the relaxation enthalpy from the non-

reversing signal (or the imaginary part of the complex heat capacity) can easily be determined after calibrating the heat-flow signal by means of synthetic sapphire.

This software extension for temperature-modulated DSC is now available for all DSC 204 F1 *Phoenix*[®] instruments for Europe, Asia (except for Japan), Australia, Latin America, South America, Africa and the Near and Middle East.

For further information, please contact your nearest NETZSCH representative.

Erwin Kaisersberger

STA-MS - with Full Steam Ahead!

Instruments for Simultaneous Thermal Analysis (STA), i.e. for simultaneous measurement of TG and DSC signals on one sample, play a decisive role in many thermo-analytical laboratories. Temperature stabilities in different gas atmospheres or phase transitions can, for example, be investigated. Simultaneous detection of the gases evolving from the sample (e.g. by mass spectrometry) allows for additional chemical analysis and finally yields a better understanding of the reaction processes.

This STA-MS coupling is now also available for humid sample atmospheres; in other words, in combination with water vapor. Typical application fields of this coupling are corrosion and scaling processes on steels, where the decarbonisation by means of the water vapor is especially important. The

same applies to the study of sintering processes in ceramic components and water gasification of petroleum coke. For the latter, the generation of hydrogen is of particular interest. Also inorganic building materials such as cement are often investigated in humid atmospheres.

The core of the arrangement shown in figure 1, the so-called water vapor furnace with a maximum temperature of 1300°C, is available for the NETZSCH models STA 409 PG, STA 409 CD or STA 449 C. Water vapor, the mass flow rate of which is controlled electronically, arrives into the sample chamber of the STA from the water vapor generator via a heated transfer line - optionally together with the carrier gas. At the gas outlet of the water

vapor furnace is the transfer line, also heated, to the NETZSCH mass spectrometer QMS 403 C *Aëolos*®.

spectrometer signals (applied with different scalings) enable interpretation of the mass

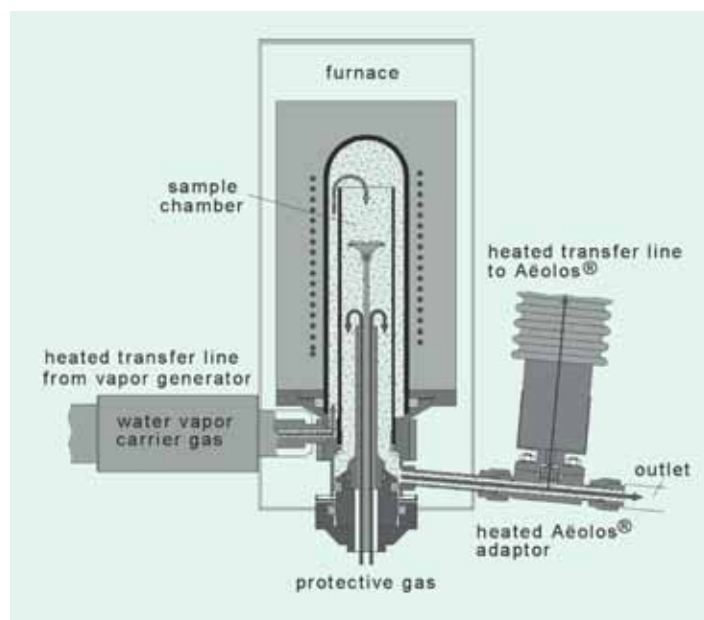


Fig. 1. STA-MS with water vapor atmosphere (schematic)

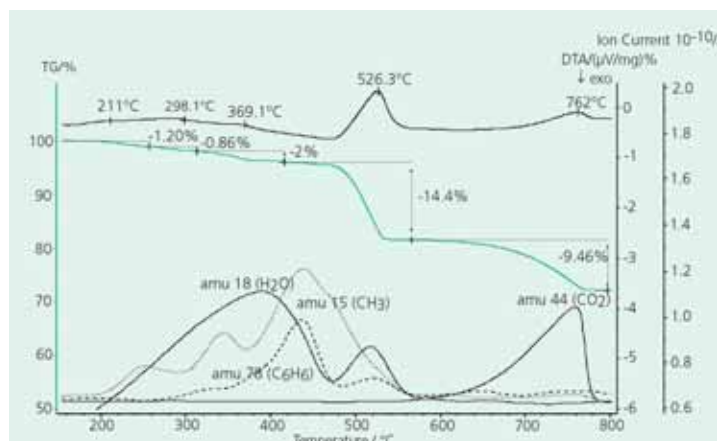


Fig. 2. Mass changes (TG), DTA signal and mass spectrometer signals of a ceramic as a function of temperature

The STA-MS results for a ceramic powder are shown in figure 2 as an example. The measurement was carried out at an absolute water vapor concentration of 34% and a heating rate of 10 K/min. Argon was used as a carrier gas; the sample weight amounted to 92.80 mg. Several mass-loss steps which are accompanied by endothermal effects can be seen over the entire temperature range. The mass

losses: in the temperature range below ~550°C, the overlapping release of water ($m/e=18$) and the release of CH_3 ($m/e=15$) and C_6H_6 ($m/e=78$) fragments occur in several steps. Between ~550°C and ~780°C, CO_2 ($m/e=44$) is detected which resulted from the decomposition of a CaCO_3 amount in the sample.

Dr. Alexander Schindler

Perfectly Combined!

The combinations of Thermogravimetry to the spectroscopic methods Fourier Transform Infrared Spectrometry (FTIR) and Mass Spectrometry (MS) have been firmly established for many years. The gases evolving are transferred to the spectrometer by means of a heated transfer line. By combining these methods, not only can it be determined which mass changes of the samples occur at which temperature, but the evolved compositions can also be identified. It is this comparatively easy access to extensive information that has greatly increased the value of coupling methods in modern analysis.

This is part of the reason why there are numerous possibilities for combining thermo-analytical instruments with spectroscopic methods such as TG-FTIR, TG-MS, TG-FTIR-MS, STA-MS, STA-FTIR and STA-FTIR-MS. This instrument family has now been extended by three additional members: DSC-FTIR, DSC-MS and DSC-FTIR-MS. While for couplings between thermogravimetry and spectroscopic

methods, decomposition behavior of the samples plays a central role, temperature programs for DSC investigations normally focus on avoiding thermal decomposition of the sample. Main applications are, for example, the comparison of sample behavior during the first and second heatings (thermoplastics), the investigation of the melting and crystallization behavior or the determination of structural changes like phase transitions (morphology). But also during these investigations, gaseous substances such as adsorbed humidity, crystal water or solvents may evolve, which can be characterized with the help of FTIR or MS.

The results of a DSC-FTIR

analysis of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) are depicted in figure 2 as an example. Between 30°C and 100°C, two overlapping endothermal effects can be

gases. As the corresponding spectra show, this is water (crystal water). In the temperature range between 130°C and 156°C, two overlapping endothermal effects also



Fig. 1. DSC 204 F1 Phoenix® with BRUKER TENSOR™ 27

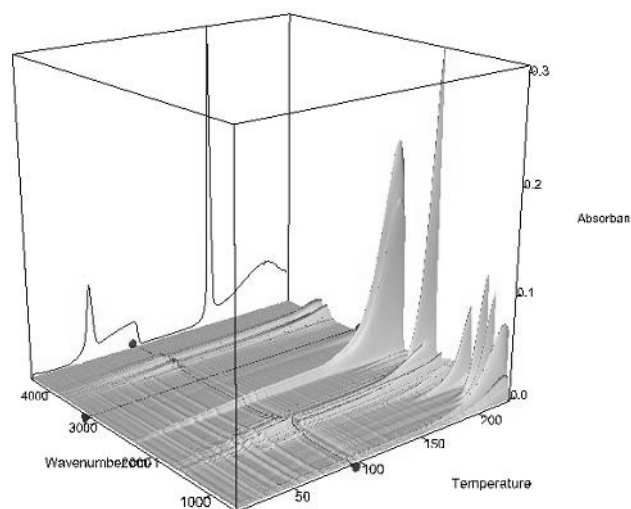


Fig. 3. Temperature-dependent presentation of all IR spectra (DSC curve shown in the vertical z-plane)

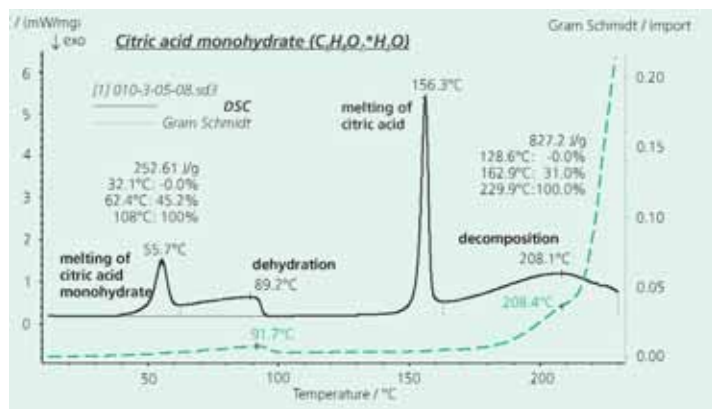


Fig. 2. Results of the DSC-FTIR analysis of citric acid monohydrate

seen in the DSC curve (black line). According to literature, the melting of citric acid monohydrate is accompanied by the release of crystal water. The experimental results show that the composition does not change until towards the end of the melting peak. At this point, the intensity of the detected IR signals slowly begins increasing due to the release of

occur. The endothermal peak with a peak temperature of 156°C is caused by the melting of the anhydrous citric acid. Immediately thereafter, decomposition starts, which is reflected in the strong increase in intensity of the detected IR adsorption bands (Gram Schmidt trace, green curve).

Dr. Ekkehard Füglein

Trade Fairs, Symposia 2005

We will be participating in the following exhibitions:

ACerS	13 -14 April, Baltimore (MD), USA
Analytica Expo 2005	19 - 22 April, Moscow, Russia
3. Int. Duroplast-tagung Iserlohn	21 - 22 April, Iserlohn, Germany
European Coatings Show	26 - 28 April, Nuremberg, Germany
Eurofillers 2005	09 - 12 May, Brügge, Belgium
Ceramics China 2005	15 -18 May, Guangzhou, China
CTAS	17 - 18 Mai, Montreal, Canada
EXPO ARPIA 2005	01 - 05 June, Buenos Aires, Argentina
21th ESAT	01 - 05 June, Jurata, Poland
IX. ECerS	19 - 23 June, Portoroz, Slovenia
ITCC/ITES	26 - 29 June, St. Andrews, New Brunswick, Canada
15. Int. Conference on Solid State Ionics	17 - 22 July, Baden-Baden, Germany
17th ECTP 2005	05 - 08 September, Bratislava, Slovakia
Chemistry-2005	05 - 09 September, Moscow, Russia
BIWIC 2005	07 - 09 September, Halle/Saale, Germany
Pharma India 2005	16 - 18 September, Hyderabad, India
NATAS	18 - 20 September, Universal City (LA), USA

New Head of Sales/Europe



Walter H. Neumann

As of April 1st, 2005, Walter H. Neumann (42) will be

responsible for "Sales Europe" at NETZSCH-Gerätebau.

With his background in chemical engineering and international business management - along with his many years' management experience in the fields of laboratory and online instruments - he is ideally qualified for this position.

You can reach Walter H. Neumann and his team under w.neumann@ngb.netzsch.com.

by the way

...on the occasion of the European Coatings Show in Nuremberg on April 27th, 2005, we will be hosting a users' seminar (in German) entitled "Thermal Analysis for the Paint and Adhesive Industries" in the "Helsinki" room in CCN East (CongressCenter Nuremberg). You are cordially invited to attend this seminar which is free of charge!

Well-known speakers will give you insight into instrument techniques and applications. A detailed program can be found on our homepage under *NEWS/Calendar of exhibitions and events*. If you have any questions or require further information, please contact Melanie Schmidt under me.schmidt@ngb.netzsch.com.

We would be happy to see you at our booth no. 221 in hall 5, where we will be represented together with our sister company NETZSCH Feinmahltechnik. There, you will be able to have a look at all of our important instruments for the determination of the properties of paints and adhesives. Apart from our classics - DMA 242 C, TG 209 F1 *Iris*[®] and DSC 204 F1 *Phoenix*[®] with automatic sample changer - a highlight for you might be our DEA 230, with which reactive systems can be investigated not only in the lab, but also in-situ.

We look forward to welcoming you!

New in Sales Poland



Dr. Krzysztof Hodor

Since October 2004, Dr. Krzysztof Hodor has been responsible for the sales of products of NETZSCH-Gerätebau GmbH in Poland.

Born in Krakow in 1972, he studied Technical Physics at the University of Mining and Metallurgy Krakow; he received his PhD in October 2002.

Before he joined NETZSCH Instrumenty, Sp.z.o.o., Krakow, he worked in the sales department of a manufacturer of medical engineering equipment.

Dr. Hodor is looking forward to providing you with support in any way he can. You can reach him under k.hodor@netzsch.com.pl.

Imprint

04/05

Publisher:
NETZSCH-Gerätebau GmbH
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Printed in Germany (4700)

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